To:

02 AUG 2001 700 09/890820

IN THE UNITED STATES PATENT AND TRADI MARK OFFICE REQUEST FOR FILING NATIONAL PHASE OF PCT APPLICATION UNDER 35 U.S.C. 371 AND 37 CF 3 1.494 OR 1.495

Asst. Commissioner of Patents Washington, D.C. 20231

TRANS	MITTAL LETTER TO THE UNITED S	STATES	Atty Dkt:	1001	38037 ga
DESIG	NATED/ELECTED OFFICE (DO/EO/	US)		M#	Client Ref.
From:	Kevin E. Joyce		Date: Au	st 2, 2001	
	This is a REQUEST for FILING a PO	CT/USA National	Phase Applicat	n based on:	
1.	International Application	2. Internation	nal Filing Date	3. Earlie	est Priority Date Claimed
	PCT/AT00/00334		nber 2000 IONTH Yea	-	ecember 1999 MONTH Year
4.	Measured from the earliest priority of filed within:	'		use it	em 2 if no earlier priority
	(a) 20 months from above item 3	date (b)	30 months fron	above item 3 dat	te,
	(c) Therefore, the due date (unexter	ndable) is Augu	st 20, 2001		
5.	Title of Invention PROCESS FOR F	PRODUCING MA	RL SLAG		
6.	Inventor(s) EDLINGER, Alfred				
Applica	ant herewith submits the following unc	ler 35 U.S.C. 371	to effect filinç		
7.	⊠ Please immediately start national	l examination pro	ocedures (35 (э.С. 371 (f)).	
8.	A copy of the International Ap English but, if in foreign language, fi				
	a. Request; b. Abstract; c. pgs. Spec. and Claims; d. sheet(s) Drawing which a	re 🗌 informal	☐ form	of size	A4
9.	☑ A copy of the International Ap	plication has be	en transmitt∈d	l by the Internati	onal Bureau.
10.			Request; (2)		
	b. is not required, as the ap	informal	☐ for a	al of size] A4 🔲 11"
	c. is not herewith, but will be Notice per Rule 494(c) if d. Translation statement at	box 4(a) is X'd o	r Rule 495(c ∍f	hcoming PTO Mis box 4(b) is X'd.	ssing Requirements

09/890820 JC05 Rec'd PCT/PTO 0 2 ARUG=20013

KE: U	SA Natio	onal Filing of PC1/ A100 / 00334
	⊠ a. ⊠	PLEASE AMEND the specification before its first line by inserting as a separate paragraph: This application is the national phase of international application PCT /AT00 /00334
	b. 🔲	filed December 11, 2000 which designated the U.S.— This application also claims the benefit of U.S. Provisional Application No.
12.		Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)), i.e., <u>before 18th month</u> from first priority date above in item 3, are transmitted herewith (file only if in <u>English</u>) including:
13.	\boxtimes	PCT Article 19 claim amendments (if any) have been transmitted by the International Bureau
14.		Translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)), i.e., of claim amendments made before 18th month, is attached (required by 20th month from the date in item 3 if box 4(a) above is X'd, or 30th month if box 4(b) is X'd, or else amendments will be considered canceled).
15.	A dec a. ⊠ b. □	laration of the inventor (35 U.S.C. 371(c)(4)) is submitted herewith
16.		ternational Search Report (ISR): s prepared by European Patent Office Japanese Patent Office Other has been transmitted by the international Bureau to PTO. copy herewith (2 pg(s).) plus Annex of family members (1 pg(s).).
17.	Interr a. ⊠ b. ☐ c.1 ☐ c.2 ☐	IPER Annex(es) in original language ("Annexes" are amendments made to claims/spec/drawings during Examination) including attached amended: Specification/claim pages # claims #
	d. 🔲	Dwg Sheets # Translation of Annex(es) to IPER (required by 30 th month due date, or else annexed
18.	Inform a. ⊠ b. ⊠ c. ⊠	amendments will be considered canceled). nation Disclosure Statement including: Attached Form PTO-1449 listing documents Attached copies of documents listed on Form PTO-1449 A concise explanation of relevance of ISR references is given in the ISR.
19.	\boxtimes	Assignment document and Cover Sheet for recording are attached. Please mail the recorded assignment document back to the person whose signature, name and address appear at the end of this letter.
20.		Copy of Power to IA agent.
21.		Drawings (complete only if 8d or 10a(4) not completed): sheet(s) per set: ☐ 1 set informal; ☐ 1 set formal of size ☐ A4 ☐ 11"
22.		(No.) Verified Statement(s) establishing "small entity" status under Rules 9 & 27
23.	Prior filed i (count	
(1)	A 2143	pplication No. Filing Date Application No. Filing Date 3/99 December 20. (2) 1999
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JORS Read FOR/FID 0 2 AUG 2001

APPLICATION UNDER UNITED STATES PATENT LAWS

Invention: PROCESS FOR PRODUCING MARL SLAG

Inventor(s): EDLINGER, Alfred

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	This is a:
	Provisional Application
	Regular Utility Application
	Continuing Application
\boxtimes	PCT National Phase Application
	Design Application
	Reissue Application
	Plant Application
	Substitute Specification Sub. Spec. filed in App. No/
	Marked Up Specification re Sub. Spec. filed/

SPECIFICATION

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Process for Producing Marl Slag

The invention relates to a process for producing marl slags and marl slag cements or mixed components for mixed cements from marl having a basicity CaO/SiO_2 of < 2.0.

When producing cement, calcareous and argillaceous stones, in particular limestone and lime marl, are used as starting materials and burned to cement clinker. The lime contained in the starting materials is completely bound to silicic alumina and optionally iron, thus forming the cement minerals usual to portland cement clinker, such as alite, belite, brown millerite and glass. In order to ensure the desired sintering to cement clinker at comparatively low temperatures of about 1350°C, relatively high-quality starting materials and, in particular, highly calcareous lime marl are required. Lime marl, which usually has a basicity of between 3 and 4 and besides lime also contains SiO_2 , Al_2O_3 and iron oxides, however, occurs in nature not only in the form of relatively high-quality and largerly pure lime marl, but rather as a common or low-quality marl in substantially higher amounts. The marl that is substantially more wide-spread characterized by basicities of between 0.8 and 2 and frequently is found also in the form of argillaceous marl. Those comparatively low-grade, yet substantially more widespread starting products in conventional cement production processes cannot be used without expensive purification and lime enrichment procedures and are available in large amounts as cheap raw materials.

Slag cements and, in particular, blast furnace slag cements likewise exhibit hydraulic properties and it has already been demonstrated that, by optimizing the slag chemistry and, in particular, by adjusting basicities and aluminate contents as well as applying special activation procedures, metallurgical slags will be improved to the extent that they correspond to a

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strength development in concrete and are at least equivalent to clinker cement.

The present invention aims to render wide-spread and cheap raw material marls having low basicities apt for economic utilization in the production of slag cements or mixed components for mixed cements while, at the same time, allowing for the adaptation of the desired product qualities to the respective requirements to a high degree. To solve this object, the process according to the invention essentially consists in that in a first process step argillaceous marl or a mixture of marl and clay having a basicity of < 2.0 is dried, preheated and calcined and that, after this, the obtained product in a second process step is melted in a separate melting furnace at higher temperatures than applied in the first process step and is granulated from the melt. Calcination is an endothermic reaction, whereas melting constitutes an exothermic reaction. Due to the fact that argillaceous marl, i.e. relatively low-grade marl, having an elevated Al_2O_3 content or a mixture of low-quality marl and clay having a basicity of below 2 is dried, preheated and calcined in a first process step, it is initially safeguarded that the high amounts of CO2 released during calcining need not be heated to the temperatures usually required in cement production for sintering. The calcination of hydrate, sulfur and carbonate compounds is, thus, carried out at comparatively low temperatures such that the large gas amounts released occur at accordingly low temperature levels, thus enhancing the thermal efficiency and hence the economy of the process. On account of the relatively low purities of the starting substances used, calcination takes place already at lower temperatures than would be the case with highly pure calcium carbonates. Due to the fact that the obtained product in a second process step is subsequently melted in a separate melting furnace at higher temperatures than in the first process step, there is the possibility to subsequently correct the composition of the melt by any means whatsoever and it is

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feasible, by appropriately granulating the melt, to provide the desired glass portion and ensure that any undesired crystallization will be largely avoided during cooling. Drying, preheating and calcining in a first process step at accordingly low temperatures, moreover, allows for the use of substantially more coarse-grained charging substances as compared to known cement clinker sintering processes such that raw material preparation and, in particular, grinding will not be required, as a rule, but the coarse charging stock merely will have to be finely broken. The process according to the invention also calls for a substantially lower heat consumption than the clinker process.

Advantageously, the process according to the invention is carried out in a manner that the first process step is realized in a suspension type heat exchanger, a rotary tubular kiln, a multiple-hearth furnace or a shaft furnace, or in a fluidized bed or cyclone preheating unit. In a particularly advantageous manner, a suspension type heat exchanger may be employed, whereby it is feasible, in particular if a rotary tubular kiln or shaft furnace is employed in the first step, to choose an even coarser particle size of the charging stock, drying, preheating and calcining of a charging material having particle sizes of, for instance, up to 40mm being readily feasible.

In a particularly advantageous manner, the second process step is carried out in a melting cyclone, a rotary tubular kiln or a hearth-type furnace, or in an iron melting oxidation reactor, whereby even the formation of foamed slag may be advantageous if a meltdown oxidation reactor is employed.

What is essential in obtaining the desired cement technological properties, after all, is the adjustment of the slag basicity of the target slag, it being advantageously proceeded in a manner that the target slag is adjusted to a basicity CaO/SiO_2 of between 0.9 and 1.85 by mixing marl and

clay. If, at the same time, an Al_2O_3 content of between 6 and 20 wt.-% is aimed at, a high-quality synthetic blast furnace slag will be obtained, such Al_2O_3 contents being obtainable in a particularly simple manner by using argillaceous marls. When using other marl qualities for the main component, the desired slag chemistry may be adjusted by the aid of bauxite, clays, flue ashes or other industrial waste substances such as, for instance, red muds, sweepings, corundum-containing grinding dusts or refractory break-offs.

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What is also essential for obtaining the desired cement technological properties is, of course, accordingly rapid cooling so as to prevent the formation of crystals. Depending on the basicity of the slag, the slag is characterized by different viscosities, wherein the slag becomes highly viscous, in particular, at basicities of above 1.4, and the formation of crystals can no longer be reliably prevented in conventional granulation processes such as, for instance, during granulation in hot water. In the context of the invention it is, therefore, advantageously proceeded in a manner that the melt at basicities of > 1.4 is sprayed into a granulator and, in particular, a vapor granulator. Spray granulation, in which the cooling of the molten droplets in most cases is effected by nozzling in water or water vapor, may be substantially improved even further in that hydrocarbon is additionally nozzled into the spray granulator. The thermal decomposition of hydrocarbon withdraws heat from the sprayed droplets at a cooling gradient of 10^4 to 10^5 K/s while simultaneously forming high-quality synthesis gas, which, as in correspondence with a preferred further development of the process according to the invention, can be burned in the first process step, because the actual heat demand for calcination arises there.

35 Since, due to the relatively low purity of the starting materials, calcination takes place at relatively low temperatures, the process according to the invention

advantageously is carried out in a manner that the first process step is carried out at temperatures of up to 950° to 1000°C, drying being effected at temperatures of from 100 to 210°C, preheating being effected at 210° to 600°C and calcining being effected at 600° to 1000°C.

In the second process step, in which the slag is melted in order to subsequently solidify under the formation of a glassy consistency, it is advantageously proceeded in a manner that operation takes place at final temperatures of between 1450° and 1550°C.

As already mentioned, the process according to the invention stands out for obviating any cumbersome raw material preparation and, in particular, for not requiring any grinding of the starting materials. Advantageously, the process according to the invention is carried out in a manner that the first process step is realized with finely broken marl having a mean particle size ranging from 20mm to 30mm.

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In order to further enhance the cement technological properties, it may be proceeded according to the invention in that by-pass dust from the production of clinker is added to the charging material. Since a slag melt is produced within the context of the process according to the invention, it is thus feasible to introduce into the melt the high alkali content contained in the clinker production by-pass dust, a thus formed product being particularly reactive and characterized by high early strengths. Clinker raw materials as are used in the conventional cement production process, as 30 a rule, are characterized by high alkali contents, and the processing of such alkali-rich raw materials, as a rule, constitutes a problem of disposal, which can be solved by the process according to the invention. Also the use of other industrial alkali-containing waste substances, for instance, those from paper and cellulose pulp production, which partially include also high portions of Al_2O_3 , as well as of

organic matter such as lignin, whose calorific value may be utilized, is advantageous.

If the melt is to be subjected to a conventional water granulation procedure, its basicity must be lowered to below 1.4, whereby in those cases the Al_2O_3 content of the target slag must be increased accordingly in order to achieve a march of strength similar to clinker, of the end product.

In the context of the process according to the invention, the use of charging materials having elevated dolomite contents has no adverse effects, either, it merely having to be taken care that the MgO portion of the charging material is adjusted to below 19 wt.-%. Up to those amounts, magnesium oxide is able to enhance the granulating capacity due to the reduced slag viscosity, which will lead to an increased portion of amorphous or glassy particles.

In the following, the process according to the invention will be explained in more detail by way of exemplary embodiments.

Example 1

A marl slag is produced of the charging materials marl and clay. The starting materials had the following directional analysis:

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Component	(왕)	Marl	Clay
Loss	on	32.5	12.5
ignition			
SiO ₂		23	46
Al ₂ O ₃		4	24
Fe ₂ O ₃	:	2	15
CaO		33	0.72
MgO		3	0.33
SO3		1	_
K20		0.5	
Na ₂ O		0.2	0.1
TiO2		0.4	1
Sum		99.6	99.7
C/S		1.4348	0.0157

The starting materials were supplemented with clinker furnace by-pass dust, whereby the early strength of the marl slag was significantly increased.

In order to adjust the slag basicity ${\rm CaO/SiO_2}$ to below 1.4 so as to apply conventional water granulation, the portions of the two components in the mixture were determined as follows with a target basicity of 1.1 having been sought:

Marl portion (x) =
$$\frac{(C/S) \cdot SiO_2 \text{ (clay)} - CaO \text{ (clay)}}{CaO \text{ (marl)} - (C/S) \cdot SiO_2 \text{ (marl)}}$$
Marl portion =
$$\frac{1.1 \cdot 46 - 0.72}{33 - 1.1 \cdot 23} = 6.48$$

20 Thus, one part of clay was mixed with 6.48 parts of marl, which resulted in a mixture of 86.6 wt.-% marl and 13.4 wt.-% clay. After this, the crude mixture for the intended marl slag had the following composition:

Marl slag				
Component	Portion (%)			
SiO ₂	37.4			
Al ₂ O ₃	9.6			
Fe ₂ O ₃	5			
CaO	41.2			
MgO	3.8			
SO3	1.2			
K20	0.6			
Na ₂ O	0.3			
TiO2	0.7			
Sum	99.8			
C/S	1.1			

After drying at 100° to 210°C, preheating at 210° to 600°C and calcining at 600° to 950°C, the dried and calcined product was drawn off a suspension type heat exchanger and introduced into a melting cyclone. Within the melting cyclone, the temperature was adjusted to between 1450° and 1550°C, whereupon the melt was granulated in hot water.

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In order to reduce the nitrogen oxide portion, it is also feasible to realize a progressive postcombustion within the suspension type heat exchanger by blowing in combustion air, thereby improving the thermal efficiency. The fuel used in the melting cyclone in an alternative process conduct, in which the melt was ejected into a spray granulator, was formed by using hydrocarbons during granulation, the cracked gas or synthesis gas formed having been used as a fuel together with air in the melting cyclone. Moreover, this method of granulation caused the iron oxide of the slag to be practically completely metallized and separated by a subsequent magnetic separation of the slag.

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Example 2

Using a rotary tubular kiln in the first process step and a hearth-type furnace in the second process step, finely broken marl having a particle size of about 25mm was used. The marl slag had the following composition:

Marl slag				
Component	Portion (%)			
SiO ₂	34.6			
Al ₂ O ₃	6			
Fe ₂ O ₃	3			
Ca0	49.6			
MgO	2			
К20	0.7			
Na ₂ O	0.3			
TiO2	0.6			
Sum	96.8			

Due to the relatively high basicity of 1.43, the slag was disintegrated by spraying into a spray granulator, whereby a high portion of fine granulates was obtained. On account of the high basicity, the high portion of fine granulates was characterized by a particularly high hydraulic activity. Such a slag having a basicity of 1.43 could no longer be successfully granulated with hot water, since the portion of fines, which is important from a cement technological point of view, was hydraulically inactivated on account of the belite crystal formation.

Claims:

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- 1. A process for producing marl slags and marl slag cements or mixed components for mixed cements from marl having a basicity CaO/SiO_2 of < 2.0, characterized in that in a first process step argillaceous marl or a mixture of marl and clay having a basicity of < 2.0 is dried, preheated and calcined and that, after this, the obtained product in a second process step is melted in a separate melting furnace at higher temperatures than applied in the first process step and is granulated from the melt.
- 2. A process according to claim 1, characterized in that the first process step is realized in a suspension type heat exchanger, a rotary tubular kiln, a multiple-hearth furnace or a shaft furnace, or in a fluidized bed or cyclone preheating unit.
- 3. A process according to claim 1 or 2, characterized in that the second process step is carried out in a melting cyclone, a rotary tubular kiln or a hearth-type funace, or in an iron melting oxidation reactor.
- 4. A process according to claim 1, 2 or 3, characterized in that the target slag is adjusted to a basicity CaO/SiO_2 of between 0.9 and 1.85 by mixing marl and clay.
- 5. A process according to any one of claims 1 to 4, characterized in that the melt at basicities of > 1.4 is sprayed into a granulator and, in particular, a vapor granulator.
- 6. A process according to any one of claims 1 to 5, characterized in that the first process step is carried out at temperatures of up to 950° to 1000°C, drying being effected at temperatures of from 100 to 210°C, preheating being effected

at 210° to 600°C and calcining being effected at 600° to 1000°C.

- 7. A process according to any one of claims 1 to 6, 5 characterized in that the second process step is carried out at temperatures of between 1450° and 1550°C.
- 8. A process according to any one of claims 1 to 7, characterized in that the first process step is realized with 10 finely broken marl having a mean particle size ranging from 20mm to 30mm.
- 9. A process according to any one of claims 1 to 8, characterized in that by-pass dust from the production of 15 clinker is added to the charging material.
 - 10. A process according to any one of claims 1 to 9, characterized in that the MgO portion of the charging material is adjusted to below 19 wt.-%.
 - 11. A process according to any one of claims 1 to 10, characterized in that spray granulation is effected using hydrocarbons as a coolant and that the synthesis gas formed is burned in the first process step.

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Abstract:

Process for Producing Marl Slag

In a process for producing marl slags and marl slag cements or mixed components for mixed cements from marl having a basicity CaO/SiO₂ of < 2.0, it is provided that in a first process step argillaceous marl or a mixture of marl and clay having a basicity of < 2.0 is dried, preheated and calcined and that, after this, the obtained product in a second process step is melted in a separate melting furnace at higher temperatures than applied in the first process step and is granulated from the melt.

FOR UTILITY/DESIGN CIP/PCT NATIONAL/PLANT ORIGINAL/SUBSTITUTE/SUPPLEMENTAL DECLARATIONS

Post Office Address (include Zip Code)

RULE 63 (37 C.F.R. 1.63) DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION IN THE UNITED STATES PATENT AND TRADEMARK OFFICE



As a below named inventor, I hereby declare that my residence, post office address and citizenship are as stated below next to my name, and I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the INVENTION ENTITLED PROCESS FOR PRODUCING MARL SLAG the specification of which (CHECK applicable BOX(ES)) A. X is attached hereto. B. was filed on as U.S. Application No. BOX(ES) C. XI was filed as PCT International Application No. PCT/ AT00 /00334 2000 December and (if applicable to U.S. or PCT application) was amended on I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above. I acknowledge the duty to disclose all information known to me to be material to patentability as defined in 37 C.F.R. 1.56. I hereby claim foreign priority benefits under 35 U.S.C. 119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT International Application which designated at least one other country than the United States, listed below and have also identified below any foreign application for patent or inventor's certificate, or PCT International Application, filed by me or my assignee disclosing the subject matter claimed in this application and having a filing date (1) before that of the application on which priority is claimed, or (2) if no priority claimed, before the filing date of this application: **Priority Claimed** Date first Laid-**Date Patented** PRIOR FOREIGN APPLICATION(S) <u>No</u> Day/MONTH/Year Filed open or Published or Granted Yes Country Number χ 20 December 1999 A2143/99 Austria I hereby claim domestic priority benefit under 35 U.S.C. 119(e) or 120 and 365(c) of the indicated United States applications listed below and PCT international applications listed above or below and, if this is a continuation-in-part (CIP) application, insofar as the subject matter disclosed and claimed in this application is in addition to that disclosed in such prior applications, I acknowledge the duty to disclose all information known to me to be material to patentability as defined in 37 C.F.R. 56 which became available between the filing date of each such prior application and the national or PCT international filing date of this application: **Priority Claimed** PRIOR U.S. PROVISIONAL, NONPROVISIONAL AND/OR PCT APPLICATION(S) pending, abandoned, patented Yes No Day/MONTH/Year Filed Application No. (series code/serial no.) Х _PCT/AT00/00334 11 December 2000 pending hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and then that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon. 10 And I hereby appoint Kevin E. Joyce [Reg. No. 20508], P.O. Box 1750, Edgewater, Maryland 21037-7750, telephone number [301] 651-4946, [to whom all communications are to be directed] my attorney to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith and with the resulting patent, and I hereby authorize him to act and rely on instructions from and communicate directly with the person/assignee/firm/organization who/which first sends this case to him and by whom/which I hereby declare that I have consented after full disclosure to be represented unless/until I instruct the above attorney in writing to the contrary 19 June 2001 Date: (1) INVENTOR'S SIGNATURE: Family Name First Middle Initial Austria Bartholomaberg Austria Residence Country of Citizenship State/Foreign Country 🗸 City Dälmaweg 13, A-6780 Bartholomäberg Post Office Address (include Zip Code) Date: (2) INVENTOR'S SIGNATURE: Family Name Middle Initial First Residence State/Foreign Country Country of Citizenship City

(FOR ADDITIONAL INVENTORS, check box ____ to attach PAT 116-2 same information for each re signature, name, date, citizenship, residence and address.)